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CANADIAN PATENT

⑮ ⑯ METHOD OF SUPPRESSING THE ADHESIVE POWER OF PAINTS,
SIZES AND PRIMERS

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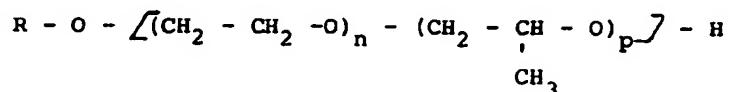
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No. OF CLAIMS 15 - No drawing

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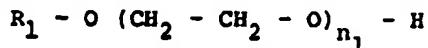
The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of substantially suppressing the adhesive power of paints, sizes or primers and of dispersing the same, which comprises bringing said paints, sizes or primers in contact with an aqueous alkaline deactivating medium containing a polyether compound having the formula:



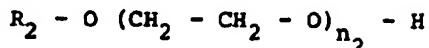
in which R is an alkyl, aryl-alkyl, aryl or alkyl-aryl, with at least 10 carbon atoms, and n and p are integers one of which can be 0, the sum n + p being at least equal to 12, said alkaline medium consisting of an alkaline aqueous solution of said polyether having a pH up to 12.

2. A method according to claim 1, wherein the polyether has the formula:



in which the group R_1 is an alkyl radical and n_1 is at least equal to 16.

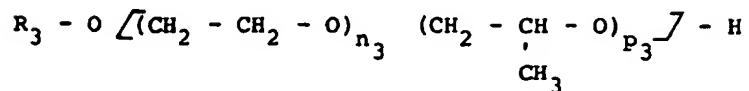
3. A method according to claim 1, wherein the polyether has the formula:



in which R_2 is an alkyl-phenyl group and n_2 is greater than 12.

4. A method according to claim 3, wherein in said polyether the R_2 radical is an octylphenyl, an isooctylphenyl or a nonylphenyl group.

5. A method according to claim 1, wherein said poly-ether has the formula:



in which R_3 is an alkyl group and the sum of the indices $n_3 + p_3$, both other than 0, is at least equal to 12.

6. A method according to claim 1, wherein said poly-ether contains in its molecule between about 20 and about 100 glycolic ether groups.

7. A method according to claim 1, wherein the concentration of polyether in said alkaline aqueous solution is from about 1 and about 5 g/l.

8. A method according to claim 7, wherein the concentration of polyether in said alkaline aqueous solution is from about 1 to about 3 g/l.

9. A method according to claim 1, wherein said alkaline medium comprises an alkaline agent selected from the group consisting of a hydroxide, a silicate, a phosphate, a polyphosphate, a borate, a perborate and a carbonate of an alkali metal.

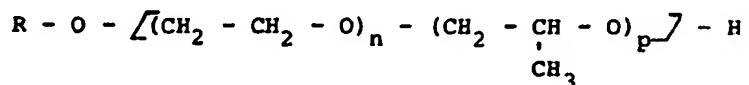
10. A method according to claim 9, wherein the alkaline agent concentration is from about 2 to about 5 g/l.

11. A method according to claim 10, wherein the alkaline agent concentration is of about 3 g/l.

12. A method according to claim 1, wherein the aqueous solution contains up to about 1000 ppm of an anti-foaming agent consisting of fatty esters of vegetal origin or of mineral oils.

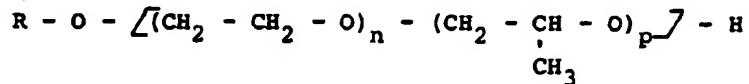
13. A method according to claim 1, wherein the processed paint, size or primer dispersed in said solution is further allowed to settle to enable the removal thereof from the solution which may be recirculated.

14. A method of preventing formation of paint, size or primer deposits on the walls and bottom of paint booths during use, which comprises applying to said walls and bottom an aqueous alkaline solution of a polyether compound having the formula:



in which R is an alkyl, aryl-alkyl, aryl or alkyl-aryl group, with at least 10 carbon atoms, and n and p are integers of which one can be 0, the sum n + p being at least equal to 12, said alkaline solution having a pH up to 12.

15. A method for deactivating paints, sizes or primers dispersed in water solution, which comprises adding to said solution up to 5 g/l of an alkaline agent and a polyether of the general formula:



in which R is an alkyl, aryl-alkyl, aryl or alkyl-aryl group, with at least 10 carbon atoms, and n and p are integers of which one can be 0, the sum n + p being at least equal to 12.



This invention relates to a method of suppressing the adhesive power of paints, sizes and primers. More particularly, it relates to a method of preventing formation of paint, size or primer deposits on the walls and bottom of paint booths during use, especially painting cabins for automobile bodies.

It is known that paint is generally applied by spraying on to automobile bodies which are passed through these cabins, this type of application being hence accompanied by appreciable losses of paint, among other things by deposition on the walls of such cabins.

These deposits must be continuously removed if it is desired to avoid frequent interruptions of the operation of the cabin, which have necessitated long and fastidious cleaning, especially by scraping, of the walls of the cabin. To prevent the formation of these deposits, recourse has been had to trickling over the walls of the cabin, a liquid composition, especially alkaline in nature, containing ingredients adapted to deactivate the paint, particularly to reduce the adhesive power of the paint particles, to entrain them into channels for their removal and/or tanks or vats positioned below the cabins.

As far as possible separation of the abovesaid liquid composition is effected inside these tanks and it can then be circulated. Particles of paint are entrained in the form either of a foam which floats on the surface of the liquid composition or of a mass which precipitates to the bottom of the tank.

Various types of alkaline deactivating compositions have already been proposed, containing among other things organic solvents, such as aliphatic or aromatic hydrocarbons or other ingredients, for example alkyl-ketones, in the presence of non-ionic wetting or emulsifying agents, among which are included condensation products of ethylene oxide and alcohols or alkyl phenols, these condensation products being representable by the



general formula $X-O-[CH_2-CH_2-O]_nH$ in which X is an alkyl or alkyl-aryl group, the index n being an integer which must however remain fairly low, particularly not exceed 8 and preferably remain below this value, to avoid difficulties resulting from the foaming power of these condensation products, which, as has been observed with those condensation products of the type whose use has already been proposed, increases with the value of the index n .

It has however been found, in practice, that known
10 liquid deactivating compositions, are not entirely satisfactory.

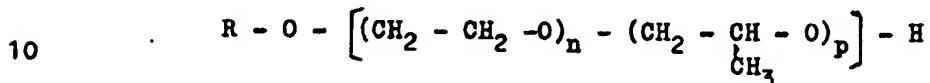
In fact, the adhesive power of the paint is not totally destroyed, so that particles of paint adhere to the walls of the spraying cabin and gradually form deposits having a particularly high cohesion. Thus it becomes extremely difficult to detach them without having recourse to improved mechanical means, requiring a stoppage of operation for restoration of the cabin to proper condition. Besides, those of the particles which are entrained have too greater tendency to remain in suspension in the denaturing compositions inside the abovesaid
20 tanks, so that the desired separation inside the latter is often found to be impractical. This necessitates a constant supply to the cabins of fresh deactivating compositions.

Moreover known deactivating liquid compositions are generally only suitable for the elimination of certain types of paint, so that the use, in a cabin, of another type of paint, must then necessarily be accompanied by a change in the nature of the compositions used.

It is an object of the invention to overcome the difficulties which have been indicated above. It is a particular
30 object of the invention to provide an improved method of suppressing the adhesive power of paints, sizes or primers currently used in industry, whereby it is unnecessary to modify the deactivating

composition utilized each time that the type of paint used is changed.

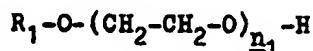
In accordance with the invention, there is thus provided a method of substantially suppressing the adhesive power of paints, sizes or primers and of dispersing the same, which comprises bringing the paints, sizes or primers in contact with an aqueous alkaline deactivating medium containing a polyether compound having the formula:



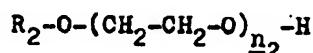
in which R is an alkyl, aryl-alkyl, aryl or alkyl-aryl, with at least 10 carbon atoms, and n and p are integers one of which can be 0, the sum n + p being at least equal to 12, the alkaline medium consisting of an alkaline aqueous solution of the polyether having a pH up to 12.

The abovesaid ethers can be obtained in a manner known in itself, especially by the reaction of the alcohol or phenol ROH from which they are derived, with the corresponding number of molecules of ethylene and/or propylene oxide.

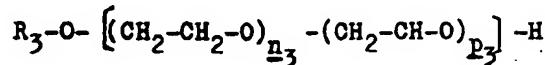
20 Groups of polyethers whose use is advantageous in the method according to the invention, are those which can be represented by one of the following formulae:



in which the group R₁ is an alkyl radical containing at least 12 carbon atoms and n₁ is at least equal to 16; or



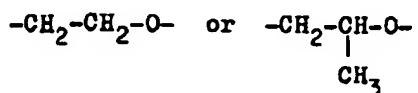
30 in which R₂ is an alkyl phenyl group, preferably octylphenyl, isooctylphenyl or nonylphenyl and n₂ is greater than 12, or



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in which R_3 is an alkyl group containing at least 10 carbon atoms and the sum of the indices n_3 and p_3 , both not zero, is at least equal to 12.

Preferably, the polyethers used contain at least 20, especially 20 to 100, glycolic ether groups of the type



10 in their molecules. In other words, the preferred polyethers result from the reaction of 20 to 100 moles of ethylene oxide per mole of the corresponding alcohol or phenol.

As regards the alkaline agents used in the formulation of the deactivating solutions utilized in accordance with the invention, recourse may be had to the usual alkaline agents, such as for example hydroxides, silicates, phosphates, polyphosphates, borates, perborates, carbonates, etc. of an alkali metal, preferably sodium, although the corresponding hydroxides or salts of other alkali metals, such as lithium, potassium, cesium and rubidium can be used. They are preferably used in 20 amounts enabling adjustment of the pH of these solutions to values of the order of 10 - 12.

Use is advantageously made of deactivating solutions containing from about 2 to about 5 g/l, for example of the order of 3 g/l of the alkaline agent and about 1 to 5 g/l, preferably of 1 to 3 g/l, of the abovesaid polyether. It is observed, especially in cases where the polyethers used contain in their molecule 20 glycolic ether groups or more, that the compositions obtained form true solutions, the polyether being in fact completely soluble in water at the indicated concentrations, because of 30 the large number of glycolic ether groups which its molecule contains.

It is noted that the foaming power of the ethers used

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does not constitute in fact an obstacle to their use in baths for the deactivating of paints, this foaming power passing more or less through a maximum for ethers containing about 20 to 25 glycolic ether groups and then diminishing rapidly when the number of glycolic ether groups contained in a molecule of ether increases even more.

It is observed also that this foaming power, which appears especially on the starting-up of installations, can be efficiently overcome by having recourse to anti-foaming agents 10 used in low concentrations, ranging especially between a few ppm and 1000 ppm, especially if care is taken to select it, either from among fatty esters based on vegetable material, which are very emulsifiable and very hydrophilic and which migrate to the water-paint interfaces, or from products based on mineral oil, of low solubility in aqueous media, which exert their effect particularly at the water-air interface.

The anti-foaming agent may be added at regular intervals of time in small amounts to the deactivating solution, rather than in a single addition which results in a considerable 20 consumption of the anti-foaming agent. The additions of these small amounts of anti-foaming agents can besides often be interrupted, after the starting-up of the installation and when the working condition has become fully continuous.

The fact that the foaming power of at least certain of the polyethers mentioned above no longer constitutes a considerable drawback with regard to their use in the formulation of deactivating solutions, must doubtless be attributed to their very high deactivating activity, compared with that, particularly mediocre, of polyethers only containing a low number of oxyethylene 30 or oxypropylene groups. It is remarkable in this regard that the strong deactivating power of these polyethers renders it unnecessary in practice to have recourse to other active

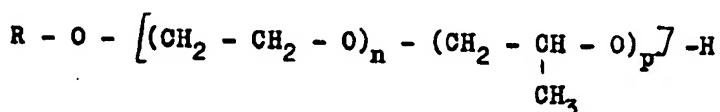
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constituents in the formulation of the deactivating solution utilized in accordance with the invention, except for the abovesaid alkaline agents.

The above solutions can be used for the deactivating all types of paint, especially automobile paint, as well as sizes or primers also used in the industry. In particular, they can be employed with success for the deactivating of glycerophthalic and acrylic paints, as well as epoxy primers, these paints and primers being representative of those which are the most difficult to deactivate.

10 These solutions not only reduce sufficiently the adhesive power of the paints to enable, especially, a continuous cleaning of painting cabins, but also enable, either a satisfactory settling of these deactivated paints in the midst of these solutions, or their retention in the foams floating on the surface of these solutions, so that the latter can be easily recirculated.

20 The invention also provides a method of preventing formation of paint, size or primer deposits on the walls and bottom of paint booths during use, which comprises applying to the walls and bottom an aqueous alkaline solution of a polyether compound having the formula:



in which R is an alkyl, aryl-alkyl, aryl or alkyl-aryl group, with at least 10 carbon atoms, and n and p are integers of which one can be 0, the sum n + p being at least equal to 12, the alkaline solution having a pH up to 12.

30 The use of anti-foaming agents at this stage is particularly advantageous, especially in the case of stable suspensions of paints, whether deactivated or not, for the

purpose, either of easily recovering the liquid medium - which can then be reused for further washings - or of facilitating their destructive treatment, to reduce their polluting power.

The subject invention will now be further illustrated with reference to the following non restrictive Examples.

Example 1

10 Into beakers of 600 ml, there are introduced 200 ml of an aqueous solution containing 0.6 g of soda and 0.2 g of polyethers identified respectively, in the Table below, by fatty alcohols or phenols and the numbers of molecules of ethylene oxide from which they have been obtained. There are then added to the beakers, with stirring, 2 g of paints also identified in the Table below, in the absence of any solvent. After standing for 48 hours, the following observations were made with respect to each of these beakers, as shown in the Table below:

- the appearance of the supernatant solution,
- the adherence of the deactivated particles and the ease of their restoration into suspension, and
- the adhesive power of these particles, after pugging or kneading.

20

This Table establishes the excellence of the results relating to the deactivating of the paints tested, and even of the primer, which is remarkable considering the well known fact that the treatment of primers is particularly difficult.

Examples 2 to 7 relate to tests carried out in experimental cabins. The anti-foaming agent used in Examples 4 to 7 is an anti-foaming agent of oily formulation, of non-ionogenic chemical character, without silicone.

Example 2

30 Tests or the deactivating of paint were carried out in an experimental cabin of 15 cm width equipped with a fan, with a washer and with a flow channel. There was trickled over

TABLE

Polyether	Concen- tration in g/l	Melamine modi- fied glycer- ophthalic base paint	Melamine modi- fied acrylic base paint	Alkyl modified epoxy base primer
<u>Product of the reaction of ethylene oxide with fatty alcohols</u>				
- Alcohol with C16.C18, 22 molecules of ethylene oxide	5 g	+++	+++	+++
	2 g	+++	+++	+++
	1 g	+++	+++	+++
- Alcohol with C16.C18, 16 molecules of ethylene oxide	5 g	++	++	++
	2 g	++	++	++
	1 g	++	++	++
- Alcohol with C10.C12, 5 molecules of ethylene oxide	5 g	++	++	++
7 molecules of propylene oxide	2 g	++	++	++
	1 g	++	++	++
- GKO alcohol with C13, 10 molecules of ethylene oxide	5 g	++	++	++
6 molecules of propylene oxide	2 g	++	++	++
	1 g	++	++	++
- Alcohol with C16.C18, 20 to 25 molecules of ethylene oxide	1 g	++	+	

Key : + + + completely deactivated paint, well dispersed, not adherent, not sticky after pugging.
 + + well dispersed paint, not adherent, slightly sticky after pugging.
 + well dispersed paint, not adherent, sticky after pugging.

TABLE Cont'd

Polyether	Concen- tration in g/l	Melamine modi- fied glycer- ophthalic base paint	Melamine modi- fied acrylic base paint	Alkyl modified epoxy base primer
<u>Product of the reaction of ethylene oxide with alkylphenols:</u>				
- Nonylphenol, 13 to 16 molecules of ethylene oxide	1 g	+++	+++	++
- Nonylphenol, 23 molecules of ethylene oxide	{ 5 g 2 g 1 g	+++ +++ +++	+++ +++ +++	+++
- Nonylphenol, 30 molecules of ethylene oxide	1 g	+++	+++	+
- Nonylphenol, 100 molecules of ethylene oxide	1 g	+++	++	+

Key : + + + completely deactivated paint, well dispersed, not adherent, not sticky after pugging.
 + + well dispersed paint, not adherent, slightly sticky after pugging.
 + well dispersed paint, not adherent, sticky after pugging.

the walls of the cabin and through the channel, a deactivating solution formed by water containing 3 g/l of soda and 1 g/l of a polyether formed by the reaction of ethylene oxide with a nonylphenol (in the proportion of 23 molecules of ethylene oxide per mole of polyether). 500 litres of this deactivating solution were employed in this test.

41.5 kg of glycerophthalic paint diluted to 23% by a solvent based on xylene and butanol were sprayed, the flow rate of the gun being about 1 kg per hour.

10 To compensate for evaporation and aspiration of water by the fan, 310 litres of water formulated with 3 g/l of soda and 1 g/l of polyether were added in amounts averaging 50 litres. The amount of anti-foaming agent added in 53 hours of operation was 0.4 litre, the greatest part of this anti-foaming agent being consumed during the period when there was no spraying of paint.

20 The result of the test was satisfactory: the paint was very well deactivated; it did not adhere to the walls of the cabin and of the channel and did not stick after kneading. It was very finely divided. A small portion of the paint was recovered at the surface with the foam; another portion was in suspension in the deactivating solution and the greatest part was deposited at the bottom of the flow channel.

Example 3

30 A test of short duration was carried out in the same experimental cabin with the same paint and the same anti-foaming agent as in Example 2, having recourse however, this time, to a polyether formed by the reaction of ethylene oxide with a mixture of fatty alcohols containing 16 and 18 carbon atoms and 22 molecules of ethylene oxide per mole of polyether. The flow channel being short-circuited, the volume of deactivating solution at 3 g/l of soda and 1 g/l of polyether was only 100 litres.

About 1.5 kg of diluted paint was sprayed in 1 h.30. The amount of anti-foaming agent added in the course of the test was 20 ml. As in Example 2, the paint was well dispersed and deposited to a great extent at the bottom of the cabin.

After standing for 60 hours, the paint retained in the foam at the surface was still in well dispersed particles; that which was in suspension in the midst of the deactivating solution is also well dispersed; that which was deposited at the bottom of the cabin was agglomerated during the period of standing in the form of a sheet which did not adhere in the least to the bottom of the cabin and which broke up easily. By kneading this paint, a non-sticky mastic was formed which easily broke into crumbs.

Example 4

A test similar to that of Example 2 was carried out in the same cabin with the same polyether (reaction product of ethylene oxide (23 molecules) with a nonylphenol). The volume of deactivating solution used was 500 litres. This time, three new paints were tested:

20 a glycerophthalic paint diluted with 21% of a heavy solvent (aromatic solvent distilling between 187 and 212°C),
an acrylic paint diluted with 22% of xylene,
a primer diluted with 10% of light solvent (petroleum fraction equals mixture of 70% of an aromatic solvent distilling between 158 and 184°C and 30% of another aromatic solvent distilling between 187 and 212°C).

A consumption of 13 ml of an anti-foaming agent in an oily formulation was sufficient in the course of 8 hours of operation of the cabin without paint spraying

30 - 16 kg of diluted glycerophthalic paint were sprayed with a gun delivery rate of 3 kg/h. The paint was well dispersed, but it stuck slightly after kneading. This is due particularly to

the fact that, the solvent being too heavy, it only evaporates very slowly, and the deactivating is ther by hind red. On the other hand, this solvent plays the role of an anti-foaming agent, so that it is not necessary to add a special anti-foaming agent to the deactivating solution.

- After emptying and renewing the deactivating solution, 10.5 kg of acrylic paint were sprayed with a gun delivery rate greater than 2 kg/h. A part of the paint floated to the surface of the solution flowing into the channel; it was well dispersed, non-adherent, but stuck very slightly after kneading. That which was deposited at the bottom of the channel was well deactivated. This paint playing to a slight extent the role of anti-foaming agent, limited the overall consumption of anti-foamer to 5 ml, in 5 hours of operation.

- After further emptying and renewing the deactivating solution, 7 kg of diluted primer were sprayed in 5 hours 15 min. Recourse was had to the same amount of anti-foaming agent as in the spraying of acrylic paint.

The primer was well dispersed; it was deposited to a great extent at the bottom of the flow channel. It did not adhere to the walls, but it was sticky after kneading immediately after spraying. On the other hand, after deposition for 15 hours, it no longer stuck at all after kneading and the deactivating was very satisfactory.

Example 5

A trial was carried out in the same cabin, with the same anti-foaming agent, the same volume of solution (500 l) and the same paints as in Example 4. However this time, the polyether, the reaction product of ethylene oxide with a nonyl-phenol, contained 30 molecules of ethylene oxide.

The glycerophthalic paint used previously was diluted with a lighter solvent (that indicated for the primer in

in Example 4). 11 kg of this paint were sprayed in 9 hours of operation. The denaturation was very satisfactory. The paint was well dispersed; it did not adhere to the walls of the cabin and of the channel; it did not stick at all after kneading.

No addition of anti-foaming agent was necessary during the spraying, the paint and its solvent playing the role of anti-foaming agent.

10 - There were then sprayed, without previous emptying of the installation, 12 kg of acrylic paint diluted with 22% of xylene, in 7 h.20 min of operation. 4 ml of anti-foaming agent were added during this time.

The paint floated to a large extent and was well dispersed. After the solution had stood for 15 hours, this paint formed a skin at the surface which was removed and broke very easily. It had the appearance of a non-sticky mastic. Its denaturation, although not complete, was very satisfactory.

Example 6

20 A test was carried out in the same cabin, with the same anti-foaming agent, but a volume of solution of 375 litres, a part of the flow channel being by-passed.

This time, the polyether, the reaction product of ethylene oxide with a nonylphenol, contained in its molecule, 100 oxyethylene groups.

6 paints were sprayed into the solution, without the latter being renewed:

3 glycerophthalic paints,
2 acrylic paints,
1 primer.

30 A few millilitres of anti-foaming agent were added when the cabin operated without spraying of paint.

An addition of 100 litres of deactivating solution

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was made in the course of the tests to compensate for losses.

It was noted that the polyether used deactivated the paints generally less well, but that it had a tendency to make the paint float, which permitted rapid removal of the latter, especially in installations comprising a surface scraper or the like.

Tests were carried out under the following conditions:

- 11 kg of glycerophthalic paint used in Example 5 and diluted in the same way were first sprayed. A large part of this paint floated on the solution in the flow channel. It was well denatured and easily recoverable by scraping.

10 - 2.750 kg of acrylic paint used in Example 5 were sprayed. The larger part of this paint also floated. Its deactivating was moderate and was sticky after kneading, but it was easily removed by scraping.

- 5.500 kg of primer used in Example 4 was sprayed. Like acrylic paint, it floated partially. It was nonetheless easily removed by scraping.

20 - 4 kg of a second glycerophthalic paint was sprayed. This paint floated also partially; it was nonetheless easily separated by scraping at the surface.

- 3.750 kg of glycerophthalic paint used in Examples 2 and 3, and diluted with 23% of a solution based on xylene and butanol, were sprayed. The paint floated to a large extent and was very well dispersed and denatured.

- 4.400 kg of a second acrylic paint were sprayed. This paint was diluted with 70% of solvent based on xylene and butanol. It floated almost entirely and was very well deactivated.

30 The consumption of anti-foaming agent was practically nil in the course of these tests with paint spraying.

Example 7

A test was carried out in a larger experimental cabin,

having a width of 1m, equipped with circular washing means and containing 9.7 m³ of water supplemented with 3 g/l of soda and 1 g/l of a polyether, the reaction product of ethylene oxide with a phenol (each molecule of this polyether containing 23 groups of ethylene oxide).

During operation of this cabin without paint spraying, the consumption of anti-foaming agent was about 3.5 ml/h/m³, the washing means being provided with a shield which reduced the formation of foam. Without the shield, 6 ml of anti-foaming 10 agent per m³ and per hour were necessary to avoid the formation of any foam.

After 8 hours of operation, 600 litres of water containing 3 g/l of soda and with 1 g/l of polyether were added to compensate for losses by evaporation and suction into the fan.

The three paints used in Example 4 were sprayed successively with a spray gun at a rate of about 15 kg/h. The following paints were successively sprayed, without having recourse to intermediate emptyings and to renewals of the deactivating solution:

20 - 100.6 kg of glycerophthalic paint diluted with 20% of light solvent (petroleum fraction indicated with respect to the primer in Example 4)

- 103 kg of acrylic paint diluted with 22% of xylene,

- 81 kg of primer diluted with 10% of light solvent (same petroleum fraction as above).

The solvents and viscosities of the diluted paints are those which are actually used in industry.

In the course of spraying the glycerophthalic paint the consumption of anti-foaming agent was nil, the paint and 30 its solvent themselves forming an excellent anti-foaming agent. It was the same during spraying the primer.

The diluted acrylic paint had a moderate anti-foaming

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effect. In the course of this spraying, it was necessary to add to the activating, solution 3.7 ml of anti-foaming agent per m^3 and per hour, when the washing means was not provided with a shield.

In trials with these paints, a fairly small amount of deactivated paint floated to the surface of the solution. It was well dispersed and well deactivated. The solution itself contained about 6 g/l of deactivated paint in suspension.

10 The greatest portion of the paint was collected at the bottom of the tank after emptying the installation. This deposit, which resulted from the mixing of three paints, did not adhere at all to the bottom of the tank and could be removed by means of a simple water jet. It was slightly sticky after kneading.

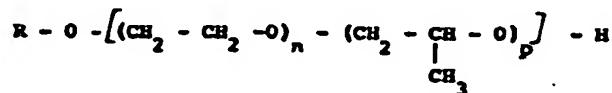
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ABSTRACT OF THE DISCLOSURE:

A method of substantially suppressing the adhesive power of paints, sizes or primers and of dispersing the same. The method comprises bringing the paints, sizes or primers in contact with an aqueous alkaline deactivating medium containing a polyether compound having the formula :



in which R is an alkyl, aryl-alkyl, aryl or alkyl-aryl, with at least 10 carbon atoms, and n and p are integers one of which can be 0, the sum n + p being at least equal to 12, the alkaline medium consisting of an alkaline aqueous solution of the polyether having a pH up to 12. The method is particularly applicable for cleaning painting cabins for automobile bodies.